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An ultrahigh-vacuum goniometer for *in situ* soft X-ray standing-wave analysis of semiconductor surfaces

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An ultrahigh-vacuum goniometer was developed for *in situ* X-ray standing-wave (XSW) analysis of semiconductor surfaces prepared by molecular-beam epitaxy (MBE). Although two ultrahigh-vacuum motors for χ and φ rotating axes are inside the analysis chamber, low-energy photoelectrons can still be collected as the magnetic field is sufficiently suppressed by using metal shields. Furthermore, the sample can be annealed at temperatures higher than 870 K on the goniometer in the analysis chamber. This goniometer is used at beamline 1A (BL-1A) at the Photon Factory, where both monochromated soft X-rays and UV radiation are available. This analysis system was shown to be suitable not only for *in situ* soft-XSW and X-ray absorption near-edge structure (XANES) studies but also for synchrotron radiation photoelectron spectroscopy (SRPES) studies. The annealing effects on an S-adsorbed GaAs(001) surface could be studied by SRPES, XANES and XSW using this new goniometer.

Keywords: goniometers; X-ray standing waves; X-ray absorption near-edge structure (XANES); photoelectron spectroscopy; semiconductor surfaces.

1. Introduction

The X-ray standing-wave (XSW) technique (Batterman, 1964) has been developed into a highly accurate technique for locating the position of particular atomic species in bulk crystals, at a crystal surface or at an interface (Zegenhagen, 1993). XSW studies using synchrotron radiation soft X-rays have recently been carried out at various facilities. When soft X-rays are used as an incident beam, the larger cross sections of light-element atoms such as Si, P, S and Cl towards softer X-rays ensure higher emission intensity from monolayer-order quantities of these atoms. Photoelectrons and Auger electrons can therefore be collected with sufficient statistics and energy resolution. Thus, several surface-structure analyses utilizing back-reflection XSWs using soft X-rays (Ohta *et al.*, 1986; Woodruff *et al.*, 1987; Hashizume *et al.*, 1992) and angle-scan soft-XSW experiments (Maeyama *et al.*, 1991, 1992; Sugiyama *et al.*, 1993) have been reported.

For investigating adsorption and heteroepitaxial growth phenomena on III–V compound semiconductor surfaces, it is advantageous to prepare the samples at the synchrotron radiation facilities where the XSW measurements are to be conducted. We previously constructed an ultrahigh-vacuum XSW analysis apparatus equipped with a molecular-beam epitaxy (MBE) growth chamber for preparing various III–V compound semiconductor surfaces (Sugiyama *et al.*, 1996*a*).

In this study, we developed a new goniometer for studying chemical reactions at semiconductor surfaces. In this new apparatus, a heater unit was attached to the goniometer in order to study chemical reactions at semiconductor surfaces with annealing. Furthermore, the magnetic field induced by two stepping motors placed in a vacuum was suppressed by using metal shields, so photoelectron spectroscopy experiments could be carried out in the same analysis system. This new goniometer system is suitable not only for *in situ* XSW, X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) studies, but also for synchrotron radiation photoelectron spectroscopy (SRPES) of semiconductor surfaces.

2. Design of the new goniometer

Fig. 1 shows a photograph of the ultrahigh-vacuum goniometer developed for the XSW experiments. This newly developed goniometer system operates in a typical four-circle mode, which is similar to that of the previously developed goniometer (Sugiyama *et al.*, 1996*a*). The ω axis, which is used for scanning the incident angle, uses the rotating axis in the conventional horizontal goniometer. The ω axis is driven by a stepping motor and the angular precision of the axis is $2.0 \times 10^{-4}\,^{\circ}$ pulse^{-1}. The ω axle is inserted into the UHV chamber through a rotary feedthrough, which is differentially pumped by an ion pump to prevent air leaks during its rotation. The χ and φ axes are mounted on the ω axle as in a normal four-circle diffractometer, and they are driven by stepping motors located in the UHV chamber. The angular precisions of the χ and φ axes are 2.0×10^{-3} and $6.0 \times 10^{-3}\,^{\circ}$ pulse^{-1}, respectively. The scanning range of the χ axis is from 0 to 90°. The scanning range of the φ axis is 360°. The Mo sample holder is held in place with metal springs on the φ table to prevent movement of the sample caused by mechanical vibration. The intensity of the diffracted X-ray beam is monitored by the photocurrent of the Cu plate, which is mounted on the 2θ arm. The 2θ arm is also driven by a stepping motor.

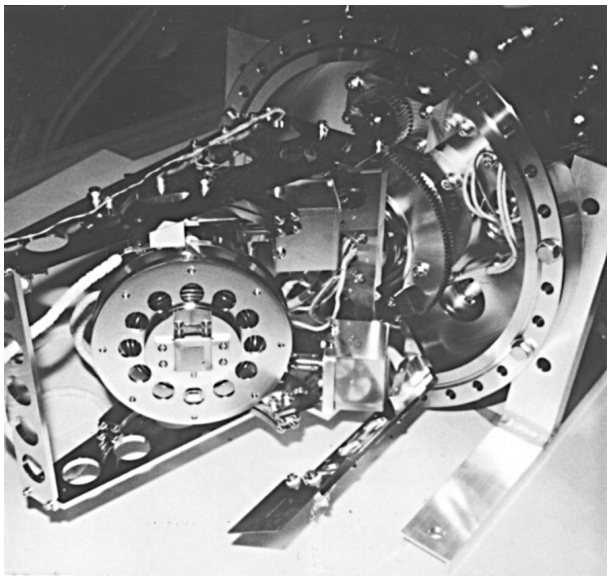

Figure 1
A photograph of the new goniometer for soft-XSW experiments.

Secondary emissions excited by the incident X-rays are collected with the detector mounted at the opposite side of the goniometer, as previously reported (Sugiyama *et al.*, 1996*a*). A high-purity Si solid-state detector (SSD) or a gas-scintillation proportional counter (GSPC) (Maeyama *et al.*, 1993) can be used for collecting fluorescent X-rays. They can be mounted at an ICF-70 flange in the polarization direction of incident synchrotron radiation soft X-rays (Sugiyama *et al.*, 1994, 1996*b*; Sugiyama & Maeyama, 1997). A hemispherical electron-energy analyser (CLAM2) for collecting Auger electrons or photoelectrons can be attached instead of the fluorescent X-ray detectors (Sugiyama, Maeyama, Heun & Oshima, 1995; Sugiyama, Maeyama, Maeda & Oshima, 1995). In order to obtain photoemission spectra in the low kinetic energy region, we used metal shields to suppress the magnetic field induced by the two stepping motors placed in the vacuum in this development.

A heater unit was attached to the new goniometer. The sample can be annealed at temperatures higher than 870 K in the analysis chamber. By using the heater unit attached to the goniometer, thermal treatments can be performed in the analysis chamber.

3. Performance

The new goniometer is used at beamline 1A (BL-1A) at the Photon Factory, where both monochromated UV radiation and soft X-rays can be used (Kawamura *et al.*, 1989). Combined analysis of an annealing process of S-adsorbed GaAs(001) surfaces was obtained by SRPES, XANES and XSW techniques in order to demonstrate the performance of the goniometer.

The S-adsorbed GaAs(001) samples were prepared by using the *in situ* S-termination process (Tsukamoto & Koguchi, 1994). The As-stabilized GaAs(001) surfaces formed by using the MBE system were exposed to S vapor (10^{-4} torr) at room temperature for 5 min. After the base pressure had recovered, the samples were transferred to the analysis chamber where the new goniometer is installed. These samples were annealed in the analysis chamber using the heater unit attached to the goniometer.

The SRPES measurements were carried out by using UV radiation of 100 eV monochromated by a 1200 lines cm^{-1} grating. As 3*d* core-level photoelectron spectra, collected with a hemispherical electron-energy analyser, from S-adsorbed GaAs(001) surfaces before and after annealing at 573 and 673 K are shown in Fig. 2. The surface-sensitive As 3*d* photoelectron spectra give the chemical states of the As atoms near the surface. Here, the As-S chemical state gradually decreases and completely disappears after annealing at 673 K for 5 min. These SRPES spectra show that photoemission spectra of the low kinetic energy region can be collected using this analysis system.

The S *K*-edge XANES measurements were obtained using synchrotron radiation soft X-rays monochromated by InSb(111) double crystals. S *Kα* fluorescent X-ray yields were collected with a high-purity Si detector with a 50 mm-thick Be window. Fig. 3 shows S *K*-edge XANES spectra, which reflect chemical state changes in adsorbed S atoms, from S-adsorbed GaAs(001) surfaces before and after annealing at 573 and 673 K.

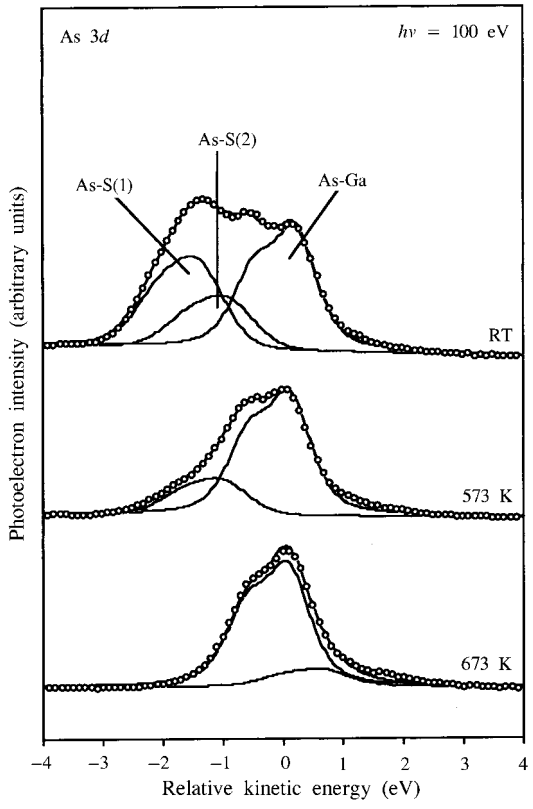

Figure 2
As 3*d* photoelectron spectra from S-adsorbed GaAs(001) surfaces before and after annealing at 573 and 673 K. The horizontal axis indicates the relative kinetic energy of photoelectrons to the As-Ga component for 673 K annealed sample. RT = room temperature.

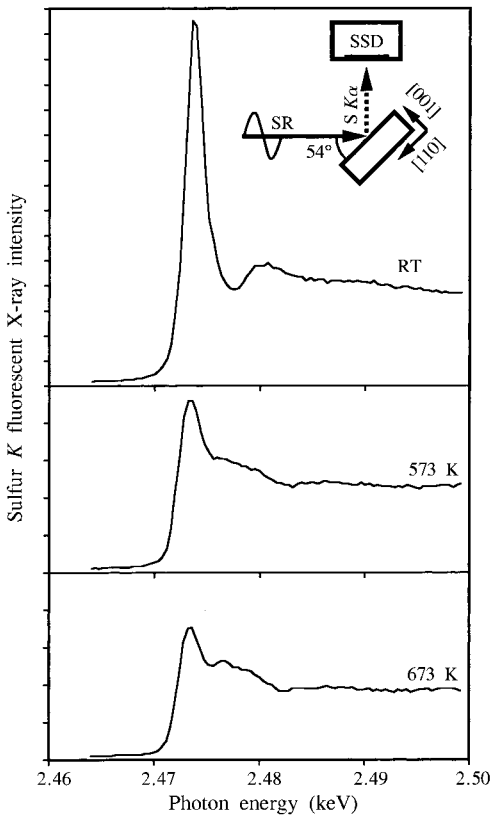

Figure 3
S *K*-edge XANES spectra from S-adsorbed GaAs(001) surfaces before and after annealing at 573 and 673 K. RT = room temperature. SR = synchrotron radiation.

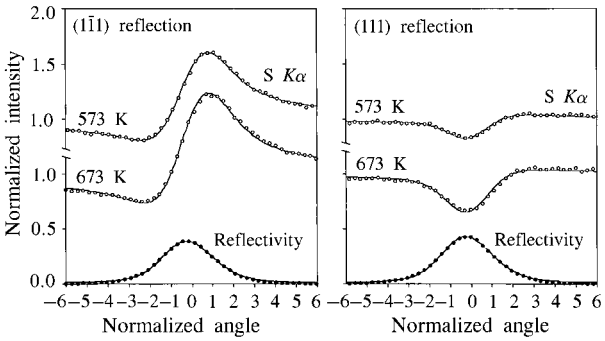

Figure 4
($\bar{1}\bar{1}\bar{1}$) and (111) soft-XSW results from S-adsorbed GaAs(001) surfaces after annealing at 573 and 673 K.

The angle-scan soft-XSW experiments were performed in order to analyse the structural changes in the S/GaAs system with annealing. Incident soft X-rays of 2.5 keV, which ensure high emission intensity from a monolayer-order quantity of S atoms, were used. The Bragg angle of the GaAs {111} reflection is 49.4° when we use photons of 2.5 keV. Fig. 4 shows ($\bar{1}\bar{1}\bar{1}$) and (111) XSW results from S-adsorbed GaAs(001) surfaces after annealing at 573 and 673 K. The incident angle was scanned near Bragg reflections. The coherent fraction F, quantitatively determined by the XSW analysis, acts as a measure of the degree of ordering. The F values are about 0.6 for the 573 K annealed sample and are improved to about 0.8 for the 673 K annealed sample.

4. Summary

In summary, an ultrahigh-vacuum goniometer was developed for *in situ* soft XSW analysis of semiconductor surfaces prepared by molecular-beam epitaxy (MBE). In this apparatus, a heater unit was attached to the goniometer for studying chemical reactions at semiconductor surfaces with annealing. Furthermore, the magnetic field induced by two stepping motors is sufficiently suppressed by using metal shields, so that photoelectron spectroscopy experiments could be carried out in the same analysis system. Combined analysis by SRPES, XANES and XSW techniques of an annealing process for S-adsorbed GaAs(001) surfaces has been demonstrated. A detailed discussion about the chemical reaction of this goniometer system will be published elsewhere. This goniometer is suitable for analysing surface chemical reactions by using synchrotron radiation.

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